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# RESEARCH MEMORANDUM

INVESTIGATION OF CERAMIC, GRAPHITE, AND CHROME-PLATED

GRAPHITE NOZZLES ON ROCKET ENGINE

By George R. Kinney and William G. Lidman

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# NATIONAL ADVISORY COMMITTEE FOR AERONAUTICS

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INVESTIGATION OF CERAMIC, GRAPHITE, AND CHROME-PLATED

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#### SUMMARY

A brief investigation was conducted regarding the use of ceramic material for rocket nozzles and the effectiveness in preventing oxidation and erosion of graphite nozzles by chrome-plating the internal surface. The investigation was conducted on a 1000-pound-thrust acid - aniline rocket. The estimated combustiongas temperatures for the runs were from 2000° to 2400° F. Nozzles were mounted in a steel housing, which was attached to the combustion chamber.

A ceramic, which contained a high percentage of sillimanite, was investigated in the form of a thin-wall nozzle (approximately 5/8 in. thick) backed with plaster of paris. The convergent section of the nozzle cracked during an initial run, but it was operated a second time without further cracking or damage. A thin chrome plating on the internal surface of graphite nozzles was effective in preventing oxidation and erosion that occurred during a run with unprotected graphite.

#### INTRODUCTION

The use of ceramics and other refractory materials for rocketengine construction is being investigated at the NACA Lewis laboratory
because such materials reduce heat transfer to the engine walls.

Desirable properties of materials to be used are high strength, low
thermal conductivity, high heat capacity, high melting point, and
good resistance to thermal shock, oxidation, and erosion. There are
ceramics that satisfy most of these requirements but have a low
resistance to thermal shock. Some grades of graphite have good
resistance to thermal shock and other desired properties but have a
low resistance to oxidation and erosion. Described herein is an
investigation of (1) the thermal-shock resistance of a ceramic
rocket nozzle containing a high percentage of sillimanite, (2) the
usefulness of a ceramic nozzle cracked during previous operation,
and (3) the effectiveness in preventing oxidation and erosion of
graphite nozzles by chrome-plating the internal surface.

# APPARATUS AND PROCEDURE

Equipment from a 1000-pound-thrust take-off assist acid - aniline rocket unit was used for the investigation. The propellant tanks, control equipment, and engine assembly were mounted on a thrust stand. The propellant-injection system provided for four pairs of impinging jets; for the design acid - aniline ratio of 1.5, the resultant direction of the impinging jets was approximately axial. For an acid - aniline ratio of 3, however, the resultant direction of the impinging jets was about 9° inward. The combustion chamber was 4 inches in diameter and 13 inches long. Chromel-alumel thermocouples were pressed against the outer surface of the nozzles at the throat position.

The operating conditions and the nozzle materials for the five runs are listed in table I. The ceramic investigated contained a high percentage of sillimanite and had an aluminum-oxide enriched glaze. The thin-wall (approximately 3/8 in. thick) ceramic nozzle was held in a plaster-of-paris support, which could be inserted into a steel housing that was directly mounted on the rocket combustion chamber (fig. 1). The graphite nozzles were machined from extruded graphite rods with a diameter of 6 inches. The construction of these nozzles was such that they could be inserted in the same steel housing as used for the ceramic nozzle. The chrome plate on the internal surface of the nozzles used for runs 4 and 5 was approximately 0.003 inch thick. The internal dimensions of all the nozzles used for the five runs were the same as those for the metal nozzles used on the take-off assist units.

## RESULTS AND DISCUSSION

The results of the nozzle-material investigation are presented in table I. The table also shows estimated combustion-gas temperatures, which were relatively low (2000° to 2400° F) for rocket application. The trends of the results, however, are significant. The estimated combustion-gas temperatures were low because the actual performance of the rocket for all the runs except run 4 was only approximately 65 percent of the theoretical performance. The combustion-gas temperatures for these runs were estimated at 45 percent of the theoretical temperatures. The low performance of the runs was attributed to the condition of the nitric acid, which had been stored for several years and was exposed to possible water dilution. The nitric acid used for run 4 had been stored only a short time and the performance for this run was approximately 85 percent of the theoretical performance. The combustion-gas temperature for this run was estimated at 75 percent of the theoretical temperature.

Ceramic nozzle. - The ceramic rocket nozzle after run 1 (16-sec operation, fuel-rich mixture, estimated gas temperature of 2000° F) is shown in figure 2. The cracking in the convergent section, which was caused by thermal shock, can be seen. No appreciable erosion at the throat and no cracking in the divergent section occurred. The same ceramic nozzle after run 2 (10-sec additional operation, near stoichiometric mixture ratio, estimated gas temperature of 2400° F) is shown in figure 3. No further cracking occurred and the condition of the nozzle was little changed by the second run. The nozzle apparently withstood the second thermal shock without further damage. The effect of further operation on erosion or cracking was not investigated. Results of a similar nature are presented in reference 1 where it was observed that a ceramic lining for a combustion chamber cracked but did not fall apart during operation. These results indicate that a ceramic rocket-nozzle liner can be successfully operated after it has cracked if it is properly supported.

The effect of cracking of the ceramic nozzle on the performance of the rocket was not accurately determined because the operating conditions of the engine and the composition of the propellants were not accurately controlled. The experiments do indicate, however, that no large effect on performance resulted from the cracking of the nozzle. The extent to which cracking of the rocket nozzle affects engine performance depends upon the location and the size of the cracks. Cracks that result in an enlargement of the nozzle throat would obviously reduce engine performance.

Properties of ceramic materials usually considered for hightemperature application are strength, melting point, thermal conductivity, heat capacity, resistance to fracture by thermal shock,
and resistance to erosion and oxidation. In the application of
ceramics for rocket-nozzle or combustion-chamber liners, strength
of the material is unimportant when the material backing the ceramic
is designed to withstand the operating pressures. Resistance to
thermal shock of ceramic materials is dependent upon strength,
coefficient of thermal expansion, modulus of elasticity at fracture,
thermal conductivity, and specific heat on a volume basis. Resistance to fracture by thermal shock is unnecessary for applications
in which proper support of the ceramic nozzle or the liner prevents
damage that would affect performance. In such cases, the number of
properties to be considered in the selection of a ceramic lining is
greatly reduced.

Graphite nozzles. - The graphite nozzle without surface plating is shown in figure 4 after run 3 (17-sec operation, near stoichiometric mixture ratio, estimated gas temperature of 2400° F). The nozzle cracked into four pieces of nearly equal size and erosion at the cracks is considerable. The surface of the graphite shows signs of oxidation and erosion in other regions, especially in the convergent section. The erosion observed near the nozzle entrance between cracks is probably caused by an excess of oxygen at those places, whereas this excess of oxygen has apparently disappeared at the throat. A local excess of oxygen could result from incomplete mixing and combustion in the combustion chamber.

The graphite nozzle with chrome-plated internal surface, which was used during run 4 (19-sec operation, fuel-rich mixture, estimated gas temperature of 2200° F), is shown in figure 5. The chrome-plated surface became pitted but protected the graphite from oxidation and erosion. No cracking of the nozzle occurred. The dark spots, which can be seen on the chrome-plated surface, are pits that contained carbon deposits from combustion.

The graphite nozzle with chrome-plated internal surface, which was used for run 5 (17-sec operation, near stoichiometric mixture ratio, estimated gas temperature of 2400° F), is shown in figure 6. The nozzle cracked at three places and was held in shape by the metal housing. The chrome-plated surface was slightly pitted but protected the graphite from oxidation and erosion. The chrome plating was less pitted on this nozzle after a more severe operation than it was on the nozzle used in run 4. The nozzle used for run 5 probably had a better plating than the nozzle used for run 4.

An X-ray-diffraction analysis was made of the plated surface of the nozzle after run 4 and carbon, chromium, and chromium oxide were present. The diffraction results thus showed that the bond between the graphite and the chromium was physical; no chemical reaction took place between the chromium and the graphite to form chromium carbide, which would have provided a desirable chemically bonded coating. An examination of the nozzles shown by figures 4 and 5 indicated, however, that the physically bonded chrome plate did protect the graphite from erosion and oxidation.

The cracking of the graphite during runs 3 and 5 is attributed to the use of extruded graphite. As the result of the extrusion process, the final graphite product is non-homogeneous and tends to be striated. These striae would cause stresses in the graphite that would in turn cause cracking and fracture when the graphite is subjected to additional stresses. The use of molded graphite will eliminate initial stresses in the material.

#### SUMMARY OF RESULTS

A brief investigation was conducted on a 1000-pound-thrust acid - aniline rocket to investigate ceramic and graphite materials for rocket-nozzle construction. The operating conditions of the rocket for the investigation resulted in relatively low combustion-gas temperatures (2000° to 2400° F) for rocket application. The trends of the following results, however, are significant.

- 1. The convergent section of a supported, thin.-wall (approximately 3/8 in. thick) ceramic nozzle containing a high percentage of sillimanite cracked during an operation of 16 seconds but the damage was not appreciable.
- 2. The ceramic nozzle, which cracked during initial operation, was used for a second operation of 10 seconds without further cracking or apparent damage to the nozzle.
- 3. A thin chrome plating on the internal surface of a graphite nozzle was effective during a 17-second operation in preventing oxidation and erosion, which occurred during a similar operation with an unprotected graphite nozzle.

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#### REFERENCE

1. Woodward, William H., and Bobrowsky, A. R.: Preliminary Investigation of a Ceramic Lining for a Combustion Chamber for Gas-Turbine Use. NACA RM No. E7H2O, 1948.

TABLE I - OPERATING CONDITIONS AND RESULTS OF NOZZIE-MATERIAL EXPERIMENTS

Run	Material	Opera- tion time (sec)	Oxidant- fuel ratio	Combus- tion pres- sure (lb/sq in. abs.)	Thrust (1b)	mated combus-	Nozzle- outer- surface tempera- ture at throat (°F)	Condition of nozzles after operation
1	Ceramic containing high per-centage of sillimenite with aluminum oxide emriched glaze	16	2.5	285	944	2000	1100	Cracked in con- vergent section, otherwise undamaged
2	Seme nozzle after run l	10	3.2	255	911	2400		Same as after run l
3	Graphite	17	3.3	250	887	2400	330	Cracked into four nearly equally sized pieces; surface eroded especially at cracks
4	Chrome-plated graphite	19	1.5	265	895	2200	310	No cracking; chrome plating protected surface from erosion; chromium surface pitted
5	Chrome-plated graphite	17	3.3	263	883	2400	140	Cracked into three nearly equally sized pieces; chrome plating protected surface from erosion; surface slightly pitted



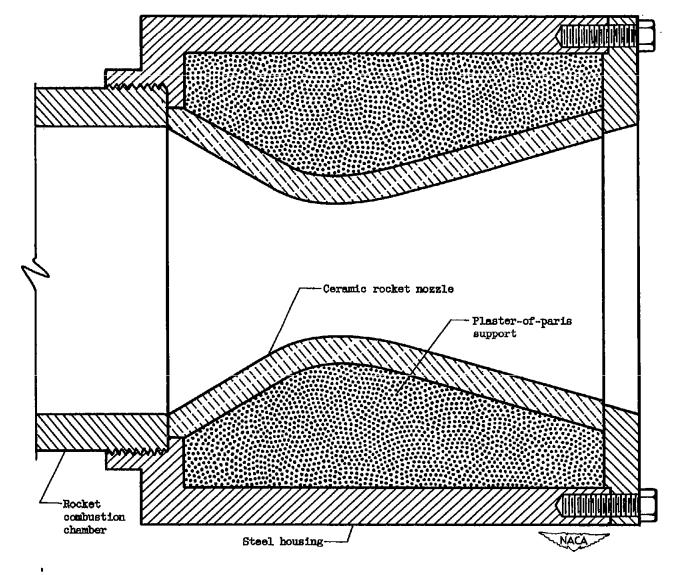


Figure 1. - Method of adapting thin-wall ceramic rocket nozzles to combustion chamber of rocket engine.

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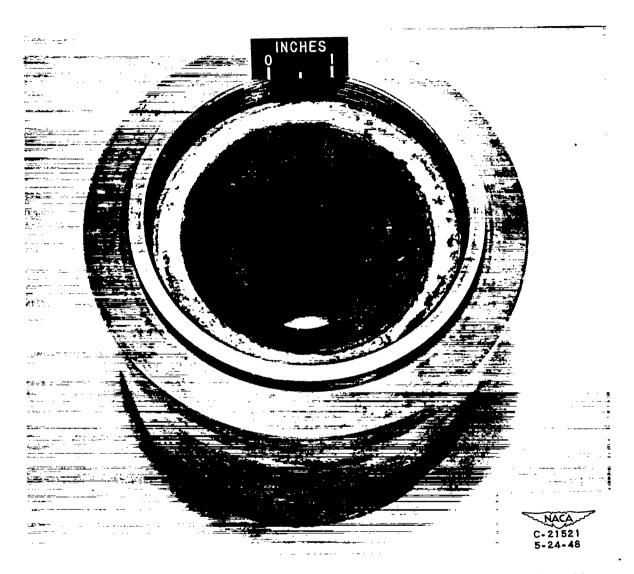


Figure 2. - Ceramic rocket nozzle after run 1 showing condition of convergent section.

Operation, 16 seconds; fuel-rich mixture; estimated gas temperature, 2000° F.





Figure 3. - Ceramic rocket nozzle (same nozzle used for run 1) after run 2 showing condition of convergent section. Additional operation, 10 seconds; near stoichiometric mixture ratio; estimated gas temperature, 2400° F.

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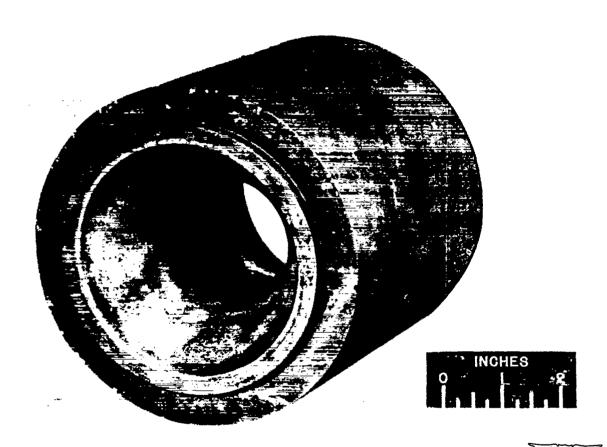


Figure 4. - Graphite rocket nozzle without surface plating after run 3 showing surface condition of convergent section. Operation, 17 seconds; near stoichiometric mixture; estimated gas temperature,  $2400^{\circ}$  F.

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NACA RM No. E8L16



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Figure 5. - Chrome-plated graphite rocket nozzle after run 4 and removal of combustion deposits from convergent section. Operation, 19 seconds; fuel-rich mixture; estimated gas temperature, 2200° F.

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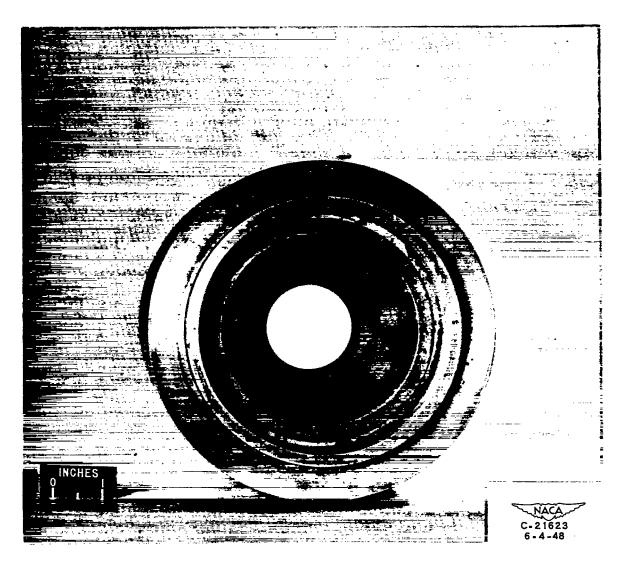


Figure 6. - Chrome-plated graphite rocket nozzle after run 5 and removal of combustion deposit from convergent section. Operation, 17 seconds; near stoichiometric mixture ratio; estimated gas temperature,  $2400^{\circ}$  F.